

|                         |                  |
|-------------------------|------------------|
| <b>COURSE CODE:</b>     | EMT 409          |
| <b>COURSE TITLE:</b>    | Soil Analysis    |
| <b>NUMBER OF UNITS:</b> | 1 Units          |
| <b>COURSE DURATION:</b> | 3 hours per week |

---

### **COURSE DETAILS:**

|                            |                           |
|----------------------------|---------------------------|
| <b>Course Coordinator:</b> | Dr. Mrs. O.O. Olayinka    |
| <b>Email:</b>              | fummy2favour@yahoo.com.ph |
| <b>Office Location:</b>    | EMT Dept COLERM           |
| <b>Other Lecturers:</b>    |                           |

### **COURSE CONTENT:**

Sampling and physico-chemical analysis of soils (including analysis of several pollutants in soil)

### **COURSE REQUIREMENTS:**

This is a compulsory course for all 400 level students in the EMT Department. It is compulsory that students should participate in all the course activities and have minimum of 75% attendance in order to be qualified to write the final examination.

### **READING LIST:**

1. Bates R.G (1954) Electrometric pH Determinations John Wiley and sons inc. New York
2. Black C.A (ed) (1965) Methods of soil Analysis Agronomy No. 9 part 2 Amer. Soc. Agronomy Madison Wisconsin
3. Jackson M.L (1962) Soil Chemical Analysis prentice Hall, New York
4. Olser, S.R. C.V, Cole, F.S. Watenable and L.A Dean (1954). Estimation of Available Phosphorus in Soils by extraction with Sodium Bicarbonate. U.S.D.A.R.939

Greweling . T. and M. \peeck (1965) Chemical Soil Tests cornel Univir. Agric. Exp. Station, Bull 1960

## LECTURE NOTES

### EXPERIMENT 1: Soil Sampling

A soil sample should be composed of several sub- samples representing a seemingly uniform area or field with similar cropping and management history.

#### Sampling Tools

There are two important requirements of a sampling tool: first, that a uniform slice should be taken from the surface to the depth of insertion of the tool, and second, that the same volume of soil should be obtained in each sub- sample.

Augers generally meet these requirements. In areas where the topsoil is dry, e.g., during summer, the topsoil sampling can be done by a metal ring, by digging out the soil inside the ring, because it is almost impossible to sample top soils with an auger.

Soil samples for micronutrient analysis should be taken using a stainless steel auger, or at least ungalvanized auger (galvanized coating is Zinc oxide).

### EXPERIMENT 2: Soil pH Determination

#### Procedure:

Soil pH in H<sub>2</sub>O (1:1 Soil water ratio).

Step 1: Weigh 20g of air- dried soil (passed through 2-mm sieve) into a 50-ml beaker.

Step 2: Add 20ml of distilled water and allow to stand for 30 minutes, stirring occasionally with a glass rod.

Step 3: Insert the electrodes of the pH meter into the partly settled suspension and measure the pH. Do not stir the suspension during measurement.

### ***EXPERIMENT 3: Determination of Organic Carbon Content of Soil***

#### ***Procedure:***

*Step 1: Weigh 1g of the soil sample (passed through 2-mm sieve) into a beaker and add 10ml of potassium dichromate solution follow by the addition of 20ml concentrated sulphuric acid. The mixture will be allowed to cool for 30 minutes.*

*Step 2: Transfer the cooled mixture into a 100ml conical flask and add 3 drops of ferroin indicator.*

*Step 3: Titrate with Ferrous ammonium sulphate solution to end point (a colour change from greenish to brown).*

*Step 4: The above processes are carried out for two blanks.*

*The titre values and the mean of the two reagent blanks (T) are recorded.*

*Calculation*

$$\% \text{ Organic Carbon} = \frac{(B-T) \times 0.5M \times 0.003 \times 100}{\text{Sample weight}} \quad \frac{\quad}{1}$$

#### **EXPERIMENT 4: Determination of Soil Particle Size**

The particle size analysis of soil estimates the percentage sand, silt and clay contents of the soil and often reported as percentage by weight of the air- dry soil. The analysis is performed on air-dried soil that is sieved with a 2mm sieve.

##### **Procedure:**

Step1: Weigh 50g of air dried soil sample (passed through 2-mm sieve) into a beaker. The soil sample is saturated with distilled water and 10ml of Calgon solution will be added for dispersing the soil sample.

Step2: Transfer the dispersed soil into a 1000ml measuring cylinder and make up to mark with distilled water. After 40seconds, the hydrometer and temperature reading will be taken and recorded.

Step3: The measuring cylinder containing the soil mixture will be allowed to stand for 2 hours. After the 2 hours, the hydrometer and temperature reading will be taken and recorded again.

##### **Calculation:**

After 40 seconds, the sand has settled and the hydrometer reading reflects the grams of silt and clay in 1 litre of the suspension. To calculate the amount of sand present in 1 litre of the suspension, the hydrometer reading at 40 seconds will be subtracted from the original weight of the soil dispersed.

$$\% \text{ Sand} = (\text{Sand weight} - \text{Hydrometer reading at 40 sec} / \text{soil weight}) \times 100$$

After 2 hours, the silt has settled. The hydrometer reading after 2 hours reflects the clay content of the soil sample.

$$\% \text{ Clay} = (\text{Hydrometer reading after 2 hours} / \text{soil weight}) \times 100$$

The silt content will be calculated by subtracting the sum of the clay and sand contents from 100.

$$\% \text{ Silt} = 100 - (\% \text{ Clay} + \% \text{ Sand})$$

#### **Experiment 5: Exchangeable Acidity in Soil (Titration Method).**

##### **Procedure:**

Step 1: Weigh 5g of air-dried soil (passed through 2-mm sieve) into a 45-ml centrifuge tube follow by the addition of 30-ml of 1N KCl . Cover the centrifuge tightly with a rubber stopper and shake for 1 hour on a reciprocal shaker.

Step 2: Centrifuge the content at 2000 rpm for 15 minutes. Carefully decant the clear supernatant into a 100ml volumetric flask.

Step 3: Another 30ml of 1N KCl will be added to the same soil sample and shake for 30 minutes. Then repeat step 2 and transfer the clear supernatant into the same volumetric flask.

Step 4: Repeat step 3 for the third time and again combine the clear supernatant into the same volumetric flask. Make up the volume to mark with 1N KCl.

#### **Titration for H<sup>+</sup> and Al<sup>3+</sup>**

Step 1: Pipette 25ml of KCl extract into a 250 ml Erlenmeyer flask (Pipette 50ml if the soil pH value is above 5.0), add approximately 100ml of distilled water.

Step 2: Add 5 drops of phenolphthalein indicator and titrate the solution with 0.05N NaOH to a permanent pink end point with alternate stirring and standing. If needed add a few more drops of indicator to replace that adsorbed by the precipitate to Al(OH)<sub>3</sub>.

Step 3: The amount of base used is equivalent to the total amount of acidity (for H<sup>+</sup> and Al<sup>3+</sup>) in the aliquot taken.

#### **EXPERIMENT 6: Determination of Exchangeable bases (K, Ca, Na and Mg). K, Na, Ca and Mg in Soil.**

##### **Procedure:**

Step 1: Weigh 5g of air dried soil sample (2mm) into a clean plastic bottle with a stopper.

Step 2: Add 100ml of 1M ammonium acetate solution into the soil. The mixture will be shaken for 30 minutes and filtered. This will be the soil extract that would be used for Na, K, Ca, and Mg determination.

Step 3: For the measurement to fall within the measurable range of the Flame Atomic Absorption Spectrophotometer, the soil extract solution will be diluted 10 times.

Step 4: Pipette 5ml of the extract into a 50ml volumetric flask, and add 1ml of lanthanum chloride solution and it will be diluted to mark with ammonium acetate extraction solution.

Step 5: These mixtures will then spray into the flame of the FAAS for the determination of K, Na, and Ca. first; the standard working solutions will be measured to calibrate the instrument.

Step 6: The soil extract solution will be diluted 17 times for the determination of Mg. To make this dilution, 3ml of the soil extract will be pipette into a 50ml volumetric flask. 5ml of SrCl<sub>2</sub> and the flask will be filled up to make with ammonium acetate extracting solution. The solution will be sprayed into the flame of the atomic absorption spectrophotometer.

#### **EXPERIMENT 7: Olsen's Test for Soil "Available" P**

**Procedure:**

Step 1: 2g of soil, 1 teaspoon of carbon black and 40ml of the extracting solution will be added into a 125 ml Erlenmeyer flask. Shake the flask for 30 minutes on a mechanical shaker.

Step2: Filter the suspension through the Whatman No 40 paper. Add more carbon black if necessary to obtain a clean filtrate. Step 3: Shake the flask immediately before pouring the suspension into the funnel. Step4: Store the solution for P determination using the colorimetric method as given in a separate section (Ascorbic acid method).

**EXPERIMENT 8: Total N in Soil (Regular Macro- Kjeldahl Method)**

**Procedure:**

Step1: Weigh 5 to 10g of soil sample containing about 10mg of N (air- dried grind to pass 0.5 mm sieve) in a dry 500ml.Macro-Kjeldjahi flask. Add 20ml of distilled water. Swirl the flask for a few minutes then allow it to stand for 30 minutes.

Step2: Add1 tablet of mercury catalyst for 1g of the  $K_2SO_4 + HgO$  mixture catalyst and 10g of  $K_2SO_4$ . Then add 30ml of conc.  $H_2SO_4$  through an automatic pipet.

Step3: Heat the flask cautiously at low heat on the digestion stand. When the water had been removed and frothing has ceased increase the heat until the digest clears. Then boil the mixture for 5hours. Regulate the heating during this boiling so that the  $H_2SO_4$  condenses about half way up the neck of the flask.

Step4: Allow the flask to cool and slowly add about 100ml of water to the flask.

Step5: Carefully transfer the digest into another clean Macro-Kjeldjahi flask (750ml). Retain all stand particles in the original digestion flask because sand can cause severe bumping during Kjeldjahi distillation. When the stand residue with 50ml of distilled water four times and transfer the aliquot into the same flask.

Step6: Add 50ml  $H_3BO_3$  indicator solution into a 500-ml Erlenmeyer flask which is then placed under the condenser of the distillation apparatus. The end of the condenser is about 4cm above the surface of the  $H_3BO_3$  solution.

Step7: Attach the 750-ml Kjedjahi flask to the distillation apparatus. Pour about 150ml of 10N NaOH through the distillation flask opening the funnel stop clock. Commence distillation.

Step8: Keep condenser cool (below 30oC) allowing sufficient cold water to flow through and regulate heat to minimize frothing and prevent suck back.

Step9: Collect 150-ml distillate and then stop distillation.

Step 10: Determine the  $NH_4-N$  in the distillate by titrating with 0.01N standard HCl for  $H_2SO_4$  using a 25-ml burette graduated at 0.1ml intervals. The colour change at the end point is from green to pink.

Step 11: Calculate the % N content in soil.

**EXPERIMENT 9 : Determination of Nitrate in Soil.**

Procedure:

Step1: Transfer a 1- ml aliquot of the soil to a vial and begin mixing. Add 0.5 ml of the brucine reagent and then add rapidly 2 ml of sulphuric acid. Mix for about 30 seconds and allow the sample to stand for 5 minutes.

Step2: Mix again, adds 2-ml distilled water and continue mixing for about 30 seconds. When working with a large number of samples, the first sample will have stood the necessary length of time by the time the sulphuric acid has been added to the last sample, whereupon continue immediately with the addition of water. Let tubes set in cold water for about 5 minutes or air- cool for 15 minutes. Measure the transmittance at 470 m $\mu$ .

**EXPERIMENT 10: Heavy Metal Determination (Cd, Zn and Pb) in Soil Sample ( Perchloric Acid Digestion)**

The heavy metal (Pb, Cd, and Zn) content in soil samples will be determined by means of the atomic absorption spectrophotometer.

Procedure

Step1: Weigh 2.0 g soil sample into a clean 125 ml Erlenmeyer flask. Add 20 ml concentrated H<sub>2</sub>SO<sub>4</sub> and 20 ml HNO<sub>3</sub>.

Step 2: Gently mix and heat at low temperature on a hot plate until dense white fumes appear.

Step 3: Allow to cool and add distilled water. Make up to 100 ml.

Step 4: Analyze for metals using atomic absorption spectrophotometer.